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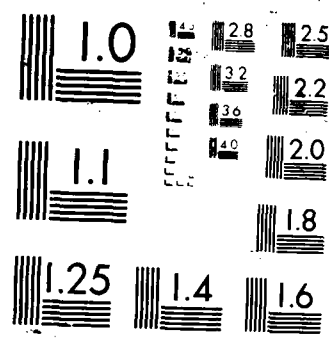
A CHEMICAL AGENT CALIBRATOR USING FLUIDIC OSCILLATOR  
FLOWMETERS(U) HARRY DIAMOND LABS ADELPHI MD  
J P GRILLS ET AL. FEB 88 HDL-TN-88-2

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<p>This paper describes the development and operation of a unique laboratory calibration instrument employing fluidic oscillators as flow-measuring devices. The fluidic oscillator is composed of a laminar proportional amplifier in a negative feedback configuration. This arrangement allows great flexibility in design because of the availability of "off the shelf" C-format laminates. It has been shown that staging such devices can increase linear ranges of operation (or turndown).</p> <p>The design goals of this instrument were to provide a highly accurate, noncontaminating, gas calibration standard to be used to calibrate gas chromatographs in the laboratory. Such a device was previously not available to the chemist and has thus advanced the state of the art for permeation devices.</p>					
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## Foreword

The purpose of this research effort is to respond to a requirement for an accurate, noncontaminating means of developing known low concentrations of chemical agents in agent-generating devices for the Chemical Research, Development, and Engineering Center (CRDEC). The current system uses as a flow source a combination of pressure regulators, rotometers, and needle valves, which are connected by plastic tubing. This configuration has the following undesirable properties:

1. Contamination from the interconnecting tubing
2. Inaccurate measurement of low flow rates
3. Insufficient flow ranges and resolution due to the limitations of the rotometers
4. Poor temperature regulation of the chemical agents before mixing

The use of fluidics will allow these obstacles to be overcome by replacing the present flow source with a monolithic device consisting of a stainless steel manifold on which stacks of fluidic laminates and flow control valves will be mounted. This in turn can be incorporated directly into the temperature-controlled oven of the existing unit.



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## 1. INTRODUCTION

Gas chromatography plays a key role in the detection of small quantities of active reagents in a large volume of relatively inert gas. This is particularly true when one desires to measure reagent amounts in the parts-per-billion range. While the chromatograph allows the detection and resolution of these small amounts, the utility of the instrument depends entirely on its being calibratable in the parts-per-billion range.

One method of calibration is to use permeation effects. One takes a sample of the reagent to be measured and encloses it in a sealed container with an opening blocked by a porous plug or a capillary tube. If a constant vapor pressure is maintained inside the container, a constant flow of reagent will leave the container. The vapor pressure is controlled by the container being enclosed in a temperature-regulated oven. The container is weighed before and after a fixed time in the oven; the difference is the loss of mass of the reagent during the elapsed time.

When the permeation flow is properly diluted, the slow rate of permeation flow permits calibration in the parts-per-billion range.

Permeation calibrators are not new and are available commercially. Before the project reported on here, permeation calibrators had poor performance because of a number of factors, among them (a) poor flow-measurement accuracy, particularly in the low ranges, (b) complete inability to measure flows in low ranges (on the order of a few milliliters per minute), (c) relatively coarse temperature control giving large vapor pressure variations, (d) poorly sealed fittings, which made it difficult and risky to calibrate dangerous reagents, and (e) large amounts of plastic and corrosive metals present in the flow paths, which gave false signatures of certain reagents to the chromatograph.

With these considerations in mind, the Harry Diamond Laboratories (HDL), under the sponsorship of the Chemical Research, Development, and Engineering Center (CRDEC), set out to design a new calibrator to reduce the previous shortcomings and improve performance by at least an order of magnitude.

## 2. GENERAL DESCRIPTION OF HDL-CRDEC CHEMICAL CALIBRATOR

As shown in figure 1, a flow diagram of the calibrator, the carrier gas enters the calibrator, passes through a heat exchanger, and then separates into two streams in the isolation chamber. One stream goes to the dilution-adjusting valve where the flow of the largest quantity of gas is controlled from 2 to 40 liters per minute (l/min). A second and smaller stream goes to the permeation-adjusting valve which controls its flow from 18 to 100 ml/min. In the dilution stream, the gas goes directly to the mixing chamber to mix with the permeation flow.

The permeation stream leaves the permeation-adjusting valve and passes through a fluidic flowmeter. It then passes through the permeation oven, which contains a reagent-charged permeation tube. This inner oven has its

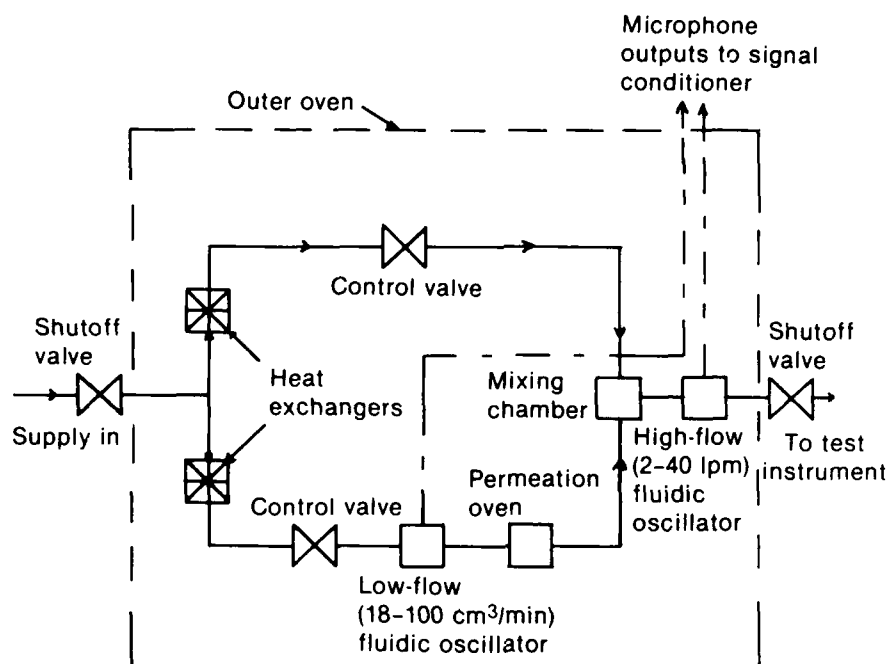


Figure 1. Block diagram showing mechanical schematic of system.

temperature controlled to a few thousandths of a degree. Within the oven, the permeation flow mixes with the reagent flow from the tube and then enters the mixing chamber.

The flow from the mixing chamber then enters the total flowmeter and exits the calibrator into the analytical instrumentation or gas chromatograph.

The components of this instrument fall into four main groups:

- (1) the thermal components, including the double oven, heat exchanger, and temperature controls,
- (2) the flow path components, including the isolation chamber, mixing chamber, inner oven exit tube, and tube fittings,
- (3) the low-flow and high-flow fluidic flowmeters, and
- (4) the electronic circuitry, which includes the microphones and amplifiers, the signal filters and conditioners, and the display circuitry.

A detailed description of each group follows.



## 2.1 Thermal Components

The heart of the thermal design is the stainless steel double oven. The outer oven has a silicone rubber resistance heater wrapped around its exterior. The temperature of this outer oven is controlled by a thermistor within the outer oven, which transmits information to a Yellow Springs model 63RC temperature controller whose specifications call for maintenance of a given set point within  $\pm 0.05^{\circ}\text{C}$ . This outer oven contains the heat exchanger, the isolation chamber, the control valves, the permeation oven, the mixing chamber, and most importantly, the fluidic flowmeters. Surrounding the outer oven is a 2-in. layer of urethane foam to provide good thermal insulation. Putting the entire flow path within the outer oven gives maximum thermal stability.

Within the outer oven, the permeation oven consists of a solid block of stainless steel shaped like an inverted "T," with both the dilution-adjusting valve and the permeation-adjusting valve as integral components of the block. Both valves have thermally isolated stems to block direct heat flow from the inner oven to the outside controls. Bolted to the block in intimate thermal contact with it are the two fluidic flowmeters, one on each side of the block. The inner portion of the permeation oven has a cylindrical cavity in which the permeation tube itself is placed. The thermal control system for the permeation oven consists of thermistors epoxied to holes in the block for good thermal contact. These thermistors provide the control signals for a Yellow Springs model 72 temperature controller which has a stated resolution of  $\pm 0.002^{\circ}\text{C}$  from a fixed set point. Three rod heaters are embedded in the block and are regulated by the temperature controller.

The major opening in the inner oven is, of course, the permeation chamber into which permeation tubes of various shapes, sizes, and materials may be placed, up to its maximum capacity of  $3/4$  in. diameter by 5 in. long.

The gas passages within this oven form a small labyrinth connecting the various components of the flow path as shown in figure 1.

The permeation oven opens for sample insertion and retrieval with a cover sealed with a gold o-ring 0.012 in. thick. The cover is welded to a coil of  $1/8$ -in. tubing which in turn is welded to the mixing chamber. Elastic deflection of the coil permits the cover to be easily displaced for sample removal.

## 2.2 Flow Path Components

The mixing chamber is of labyrinth design, incorporating twelve  $90^{\circ}$  turns in the flow path to maximize turbulence and insure adequate mixing of the dilution gas stream and the reagent-containing stream from the permeation oven. Some additional mixing occurs during the passage of the gases through the permeation oven labyrinth and the total flowmeter.

The isolation chamber is designed to minimize dynamic pressure effects due to changing flow conditions on the dilution line. A volume, large compared to equivalent lengths of tubing, is the basis of the isolation chamber. The flow entrances and exits are at right angles so that the flowing gas does not enter other openings directly. Thus, the flow variations are largely the result of static pressure effects rather than dynamic pressure ones.

The gas enters the calibrator through a heat exchanger inside the outer oven and outside the inner oven. This heat exchanger consists of two bifilar wound coils of stainless steel tubing with parallel connections to both ends of the coil for appropriate pressure drops.

Since the gas is heated during a comparatively long passage of approximately 50 ft within the heat exchanger, the temperature of the air entering the inner oven may be adjusted so that it enters the inner oven at close to the same temperature as the inner oven itself. The gas temperature is further equalized to that of the oven by passage through the block labyrinth.

The flow path is constructed entirely of stainless steel. The few internal fittings are of the Swagelok type with conical gaskets around the tubing. These fittings have shown no leakage at the moderate pressures used in the calibrator.

It is important to note that all connections in the flow path are either welded steel or compression sealed. This type of construction does not have any plastic in contact with the flow streams to give false signatures to the chromatograph, eliminating a major objection to previous calibrators.

### 2.3 Fluidic Flowmeters

The fluidic flowmeters constitute the principal element that permits flow measurement over the ranges required for calibrator operation. The dilution flowmeter is assembled using HDL's C-format laminates [1]. The unit is a three-staged device: that is, there are three active component stages which are interconnected using various transfer laminates to form a fluidic circuit. The order of assembly, or stacking order, for this circuit is listed in appendix A.

The permeation flowmeter is a single-staged device which is built of HDL X-format laminates. Since these laminates are physically smaller than C-format types, this device can measure the low flowrates (on the order of several milliliters per minute) associated with the permeation process.

The design approach used is basically the same for both flowmeters. The analysis and theory involved in this design are presented in greater detail in section 3.

## 2.4 Signal Processing

The pressure signals from the oscillators are converted to electrical signals through the use of high-performance subminiature capacitor microphones manufactured by Gentex, specifically, the Gentex model 3010. This microphone is designed to meet military standards, and its frequency response is well suited to the operating bandwidth of the fluidic devices.

The use of three-staged devices causes a considerable amount of noise to develop in the feedback line of the oscillator. This noise is found to be a function of the amount of flow passing through the system, and is made up mostly of high-frequency components. There is, however, enough noise generated within the frequency band to cause problems in transducing the signal over the entire range of oscillation. This problem is circumvented by employing both fluidic and electrical filters on the signal.

The fluidic low-pass filter consists of a capacitive volume placed at the output of the third stage of the oscillator within the feedback loop. This volume acts as an acoustic low-pass filter. Analogous acoustical and electrical schematics of such a filter are illustrated in figure 2.

Bench testing showed that an acoustical filter alone would not provide enough filtering because such a design is limited by the loading constraints imposed by the fluidic circuitry. Therefore, additional filtering of the microphone's output signal is necessary.

Initially, a fixed-bandwidth, low-pass filter was designed to further remove high-frequency components of the signal. This approach encountered problems however, because of harmonic distortion of the signal at low flow rates (corresponding to low frequencies). The next step, therefore, was to design some type of tracking filter which would automatically shift its bandwidth and track the signal as it changed frequency.

This was accomplished by implementing an Analog Devices AD534 multiplier chip configured as a voltage-controlled low-pass filter (VCLPF).

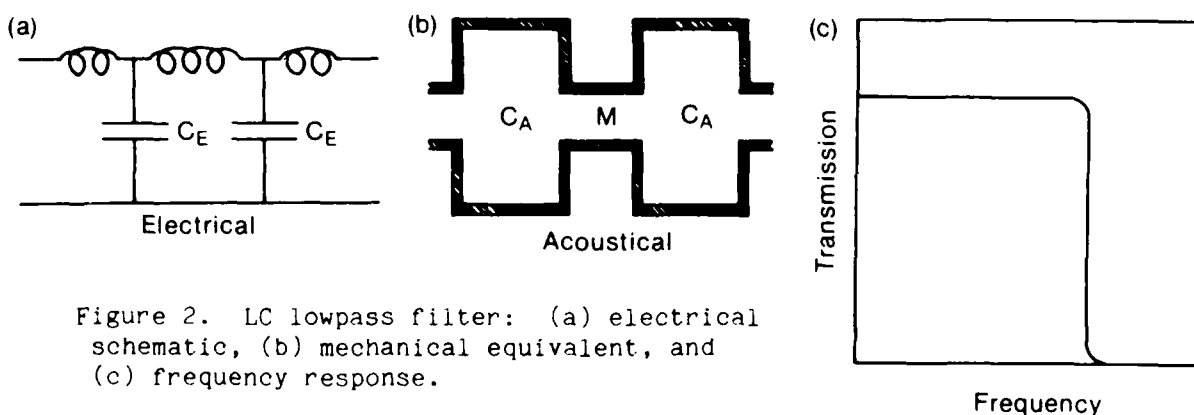
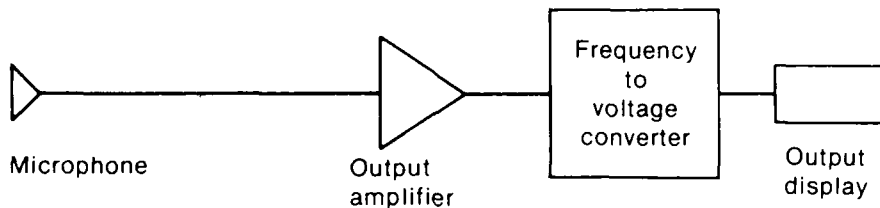


Figure 2. LC lowpass filter: (a) electrical schematic, (b) mechanical equivalent, and (c) frequency response.

The block diagram of this circuit is shown in figure 3. The control voltage is supplied by the output of the Analog Devices AD451J frequency-to-voltage converter which is used to drive the liquid-crystal displays (LCD's). The final version of this filter consisted of a four-stage circuit, whose cutoff frequency response can be seen in figure 4.

The permeation flowmeter does not have the severe problem of flow noise because of the low flowrates involved. Therefore, the fluidic low-pass filter is adequate for this signal and no electronic filtering is used.

Reagent signal:



Dilution signal:

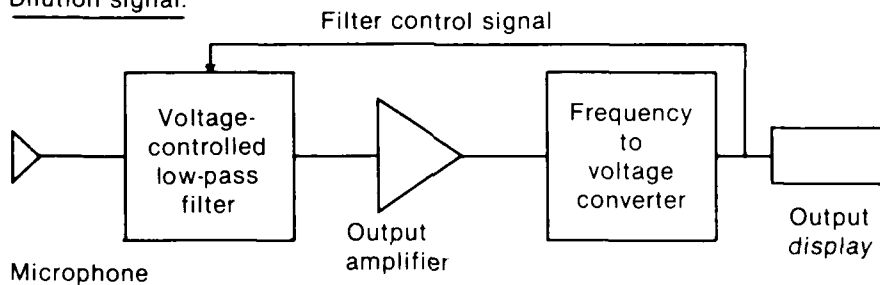


Figure 3. Electronic signal-processing block diagram.

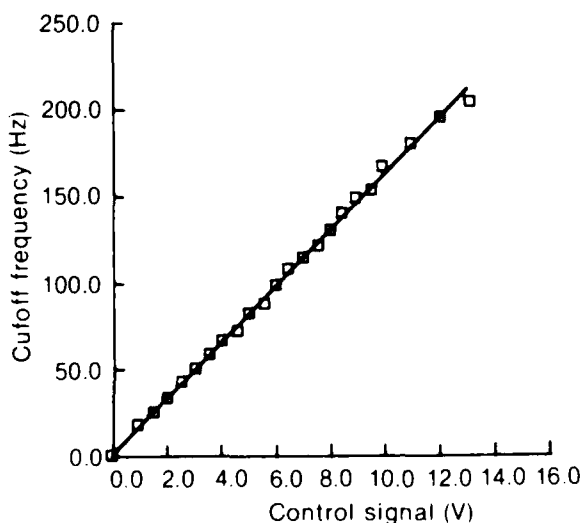


Figure 4. VCLPF frequency response.

After filtering, the signal is amplified and sent to a frequency-to-voltage converter. This electronic device has a linear relationship between input frequency and output voltage (dc). This output voltage is used to drive the LCD's and, in the case of the dilution flowmeter, it also controls the cutoff frequency of the tracking filter.

### 3. FLUIDIC FLOWMETER ANALYSIS AND DESIGN

#### 3.1 Theory of Operation

Figure 5 shows a schematic of a fluidic oscillator consisting of a C-format laminar proportional amplifier (LPA) [2-4] with feedback lines coupling the outputs with the inputs. The period of oscillation,  $\tau$ , for this device can be written as

$$\tau = 2(t_j + t_f + t_a) \quad , \quad (1)$$

where

$t_j$  = signal transport time of the supply jet from the nozzle to the splitter,

$t_f$  = time constant of the feedback channel, and

$t_a$  = acoustic time delay of the feedback channel.

The output frequency of this device is

$$f = \frac{1}{\tau} \quad . \quad (2)$$

The dominant term in equation (1) is the signal transport time,  $t_j$ . When  $t_f$  and  $t_a$  are sufficiently small, equation (2) can be approximated as

$$f = \frac{1}{2t_j} \quad . \quad (2a)$$

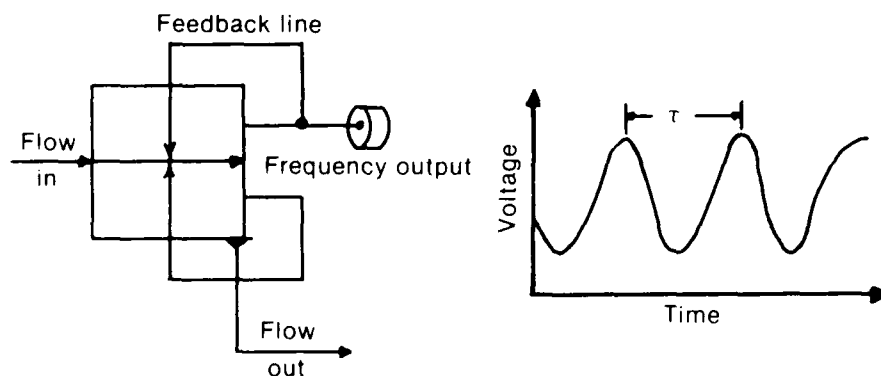


Figure 5. Single-stage oscillator circuit diagram.

The volumetric flow rate,  $Q_j$ , through the oscillator can be expressed as

$$Q_j = A_j \bar{V} , \quad (3)$$

where

$A_j$  = cross-sectional area of the amplifier nozzle and

$\bar{V}$  = average throughput velocity.

The nozzle area can be written as

$$A_j = h_s b_s = \sigma b_s^2 \quad (4)$$

where

$h_s$  = nozzle height,

$b_s$  = nozzle width, and

$\sigma$  = aspect ratio =  $h_s/b_s$ .

The signal transport time can be expressed as

$$t_j = \frac{2X_{sp}}{\bar{V}} , \quad (5)$$

where

$X_{sp}$  = nozzle-to-splitter distance.

The velocity can be expressed as

$$\bar{V} = \frac{C_1 Q_j}{A_j} , \quad (6)$$

where

$C_1$  = velocity profile constant ( $\sim 0.5$  for  $\sigma > 1$ ).

Substituting equations (4) and (6) into equation (5) gives

$$t_j = \frac{4X_{sp}\sigma b_s^2}{Q_j} . \quad (7)$$

This implies

$$f = KQ_j , \quad (8)$$

where

$$K = \frac{1}{4X_{sp}ob_s^2} \quad (9)$$

Combining equations (8) and (9) with proper units yields

$$f = \frac{521Q \text{ (l/min)}}{ob_s^3 \text{ (mm)}} \quad (10)$$

In equation (8),  $K$  is a constant independent of properties or temperature. Experimental results are shown in figure 6. When the linear relationship defined by equation (8) occurs in a device, the device is a volumetric flowmeter. However, it can be shown that as the frequency of oscillation increases, the contributions of the acoustic delay and time constant of the feedback lines increase, causing a nonlinear relationship between supply flow and output frequency. In general, state-of-the-art fluidic oscillators have at best a linear region of 10:1. In a single-amplifier configuration, the only way to maintain the predominance of the transport term ( $t_j$ ) is by making the device large and the feedback distance short. There is a fundamental limit to this reasoning, as shown by the dashed line in figure 7.

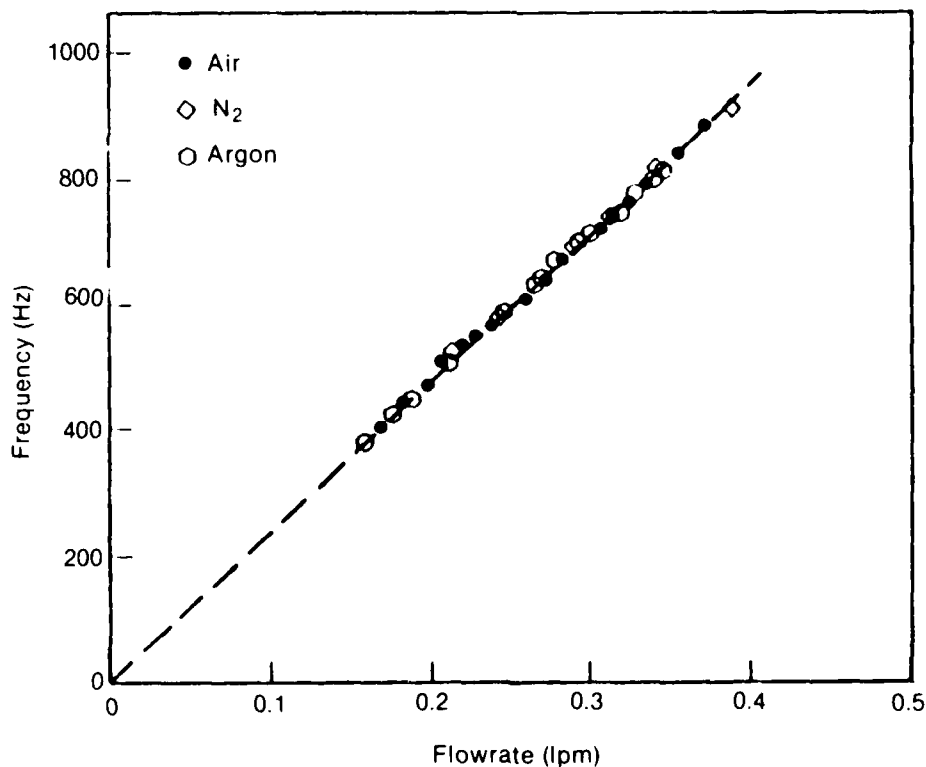


Figure 6. Volumetric flowmeter experimental data (various gases).

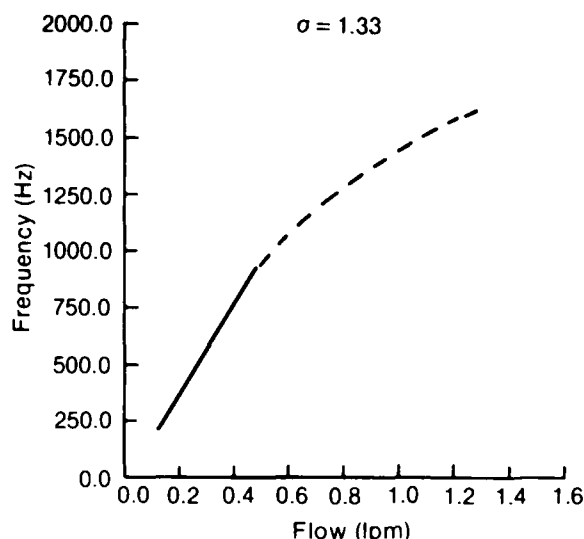


Figure 7. Nonlinearity rolloff curve (flow versus frequency for one-stage oscillator).

### 3.2 Design Considerations

One way to circumvent this problem of limited turndown range is to build multiple-staged oscillators. By following this approach, fluidic gain blocks can be constructed with multiple internal amplifiers without substantially increasing the length of the feedback lines, and by so doing, the total transport time is dramatically increased. In fact, experimental evidence has shown that the transport time may be increased by a factor of  $N$ , where  $N$  is the number of stages in a gain block. Theoretically, one might expect an  $N$ -fold increase in turndown for a volumetric flowmeter of such a configuration.

Multiple staging of amplifiers will also considerably reduce the frequency of the device (again by approximately a factor of  $N$ ). This helps overcome another limitation of a single-stage oscillator, the problem of edgetoning.

An edgetone is characterized experimentally by a sudden jump in frequency as the operating frequency approaches the edgetone frequency (see fig. 8). Several theories have been presented (Nyborg [6], Powell [7], and Meyer [8]) that attempt to explain this phenomenon. There seems to be little doubt that edgetones result from some type of sustained internal feedback in the jet region which is enhanced under certain loading conditions. This report does not go into detail on the theory of edgetones, but rather tries to explain how to go about avoiding them. Staging amplifiers within an oscillator will have the effect of decreasing the jet velocities in the said stages for a corresponding supply flow to the unit. This causes the output frequency to remain well below the edgetone frequency and to operate within the normal mode of oscillation.



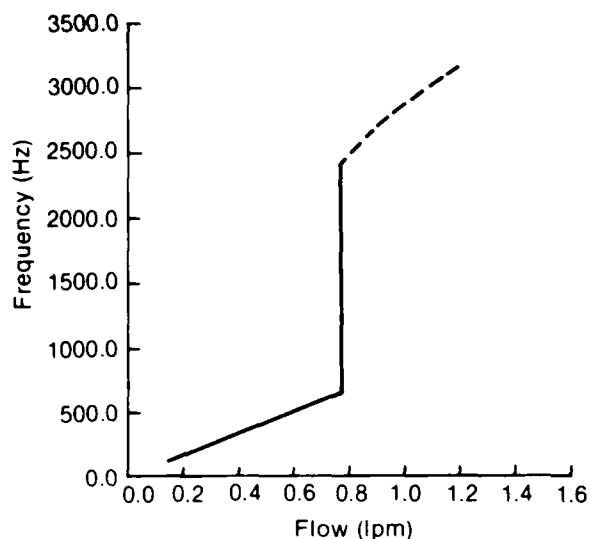


Figure 8. Edgetoning in a fluidic oscillator.

#### 4. EXPERIMENTAL RESULTS

Flow versus frequency data have been accumulated using various configurations of single- and multiple-staged oscillators. Testing of the single-stage units yielded several predictable trends. As aspect ratio and nozzle width increased, the linear region of the flow-versus-frequency curve expanded, as shown in figure 9. This observation dictated the use of  $b_s = 0.030$ -in. amplifiers (the largest available in C-format) as the preferred unit.

The simplified theory expressed by equation (10) agrees with the test results. As aspect ratio increases, equation (10) more closely follows data; this is illustrated in figure 10. This trend is due to the boundary effects on the jet's velocity profile for low aspect ratios. As aspect ratio increases, the approximation of average velocity in equations (3), (5), and (6) becomes valid.

With the information gathered by testing the single-stage oscillators used as a design guide, testing was begun on multiple-stage oscillators. A schematic of an N-stage oscillator is shown in figure 11. In an effort to keep the feedback lengths as short as possible, only odd numbers of stages were considered. Configurations employing an even number of stages necessitate cross-coupling of the feedback lines from the output of the last stage to the input of the first, which increases the total feedback length.

The idea of increasing aspect ratio to give increased linear ranges holds true for multiple-staged oscillators as well, as shown in figure 12. This approach has a fundamental limitation, however, because as aspect ratio increases, the flow needed to incite oscillation also increases. This observation gave rise to the idea of progressively increasing the aspect ratios of

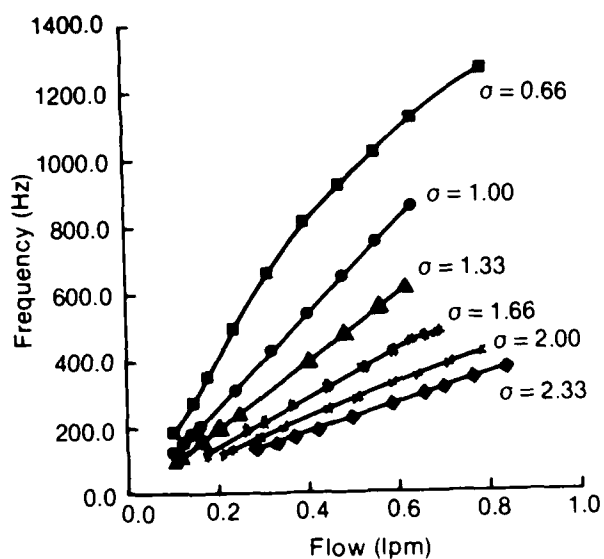


Figure 9. Single-stage curves with increasing aspect ratio and increasing linearity.

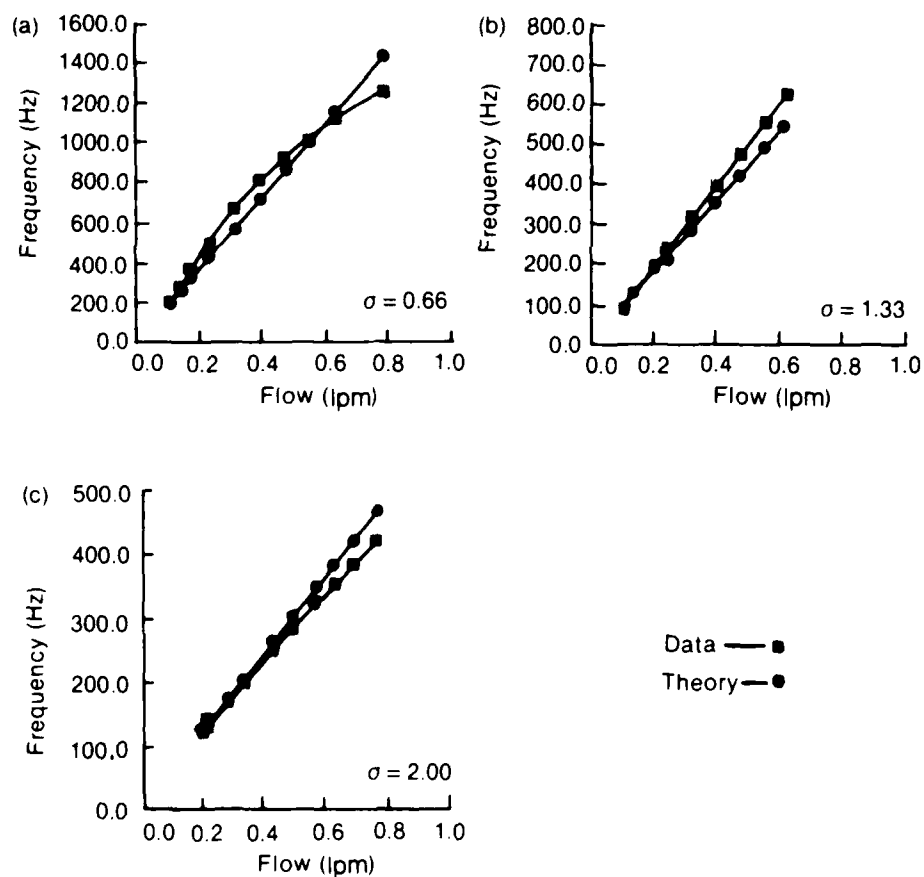


Figure 10. Data versus theory for (a)  $\sigma = 0.66$ , (b)  $\sigma = 1.33$ , and (c)  $\sigma = 2.00$ .

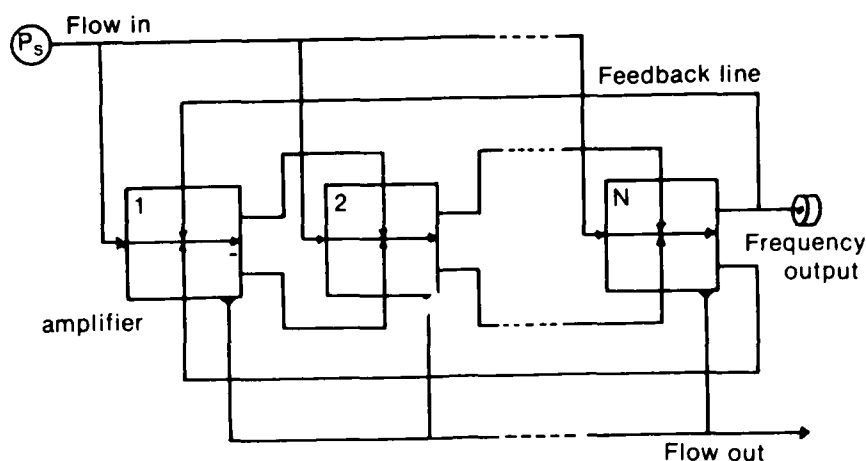


Figure 11. N-staged oscillator schematic.

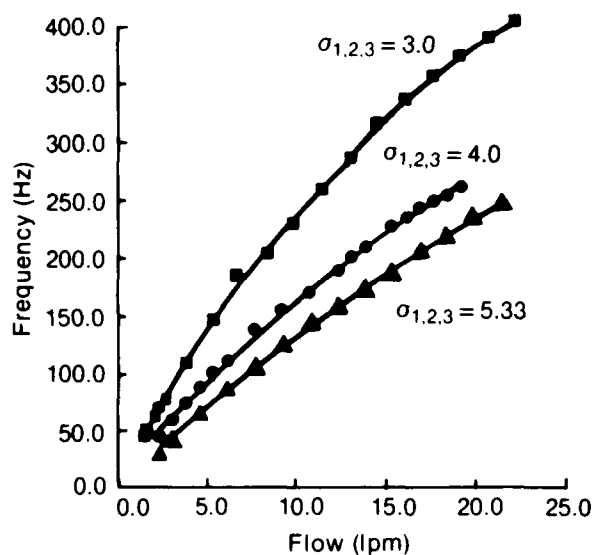


Figure 12. Multistaged oscillators with increasing uniform aspect ratio.

the separate stages internal to the multiple-staged units; i.e.,  $\sigma_1 < \sigma_2 < \sigma_3$ . This approach in fact was successful. By steadily increasing the size of the second and third stages in a three-stage oscillator, while keeping the first stage smaller, we achieved a significant increase in the linear region of the flow-frequency curve. This is shown in figure 13. The division of flow in the stack enabled the first stage to initiate oscillation at low flows, while the larger stages sustained the oscillation at higher flow rates. Using this as a design guide, we achieved a turndown of over 30:1 using a three-stage oscillator with aspect ratios of  $\sigma_1 = 3.0$ ,  $\sigma_2 = 4.0$ , and  $\sigma_3 = 8.3$  (fig. 14).

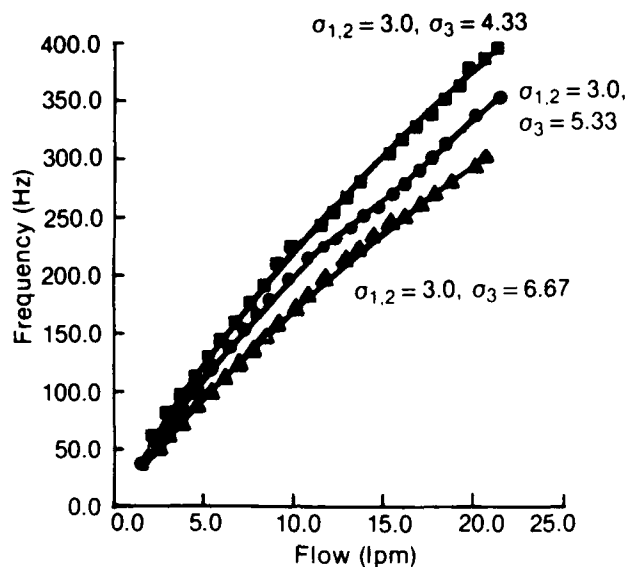


Figure 13. Three-stage oscillators with increasing aspect ratios.

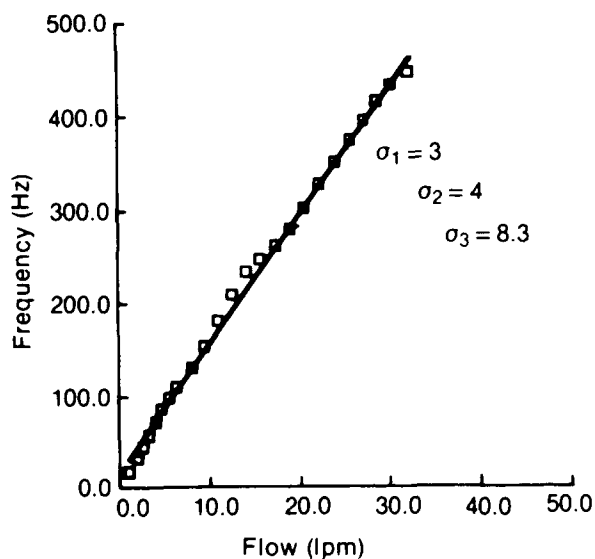


Figure 14. Three-stage flowmeter:  $\sigma_1 = 3$ ,  $\sigma_2 = 4$ , and  $\sigma_3 = 8.3$ .

## 5. PERFORMANCE TESTING OF HDL-CRDEC CALIBRATOR

In actual use, a permeation tube containing a sample of the reagent is weighed. The tube is then placed in the calibrator and a set temperature maintained in the calibrator through the controls of the calibrator. After a comparatively long period of time (up to a week), the tube is taken out of the oven and reweighed. The loss in mass divided by the elapsed time gives the mass flow rate of the reagent. It is clear that the accuracy of the instrument depends on three independent sets of measurements, namely, the loss of mass of the reagent tube, the measurement of flow rates, and the measurement of the temperature-time integral of the oven.

The loss in mass is easily measured on highly accurate chemical balances and is not a function of calibrator development.

For the fluidic flow measurement, HDL uses secondary standard resistive Vol-o-Flo flowmeters that are calibrated by the U.S. Army Primary Standards Laboratory at Redstone Arsenal, AL. These flowmeters display an accuracy of  $\pm 0.30$  percent of reading. The calibrator flow measurements were quite repeatable and displayed no measurable hysteresis within the measurement capability of the Vol-o-Flo flowmeters. The calibration curves for the two flowmeters are shown in figures 15 and 16.

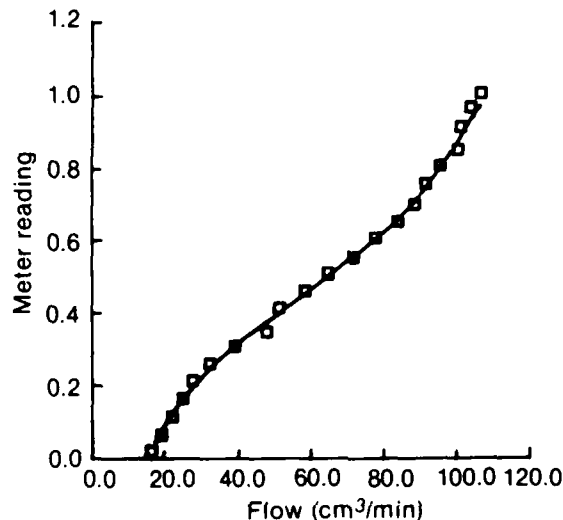


Figure 15. Permeation flowmeter calibration curve.

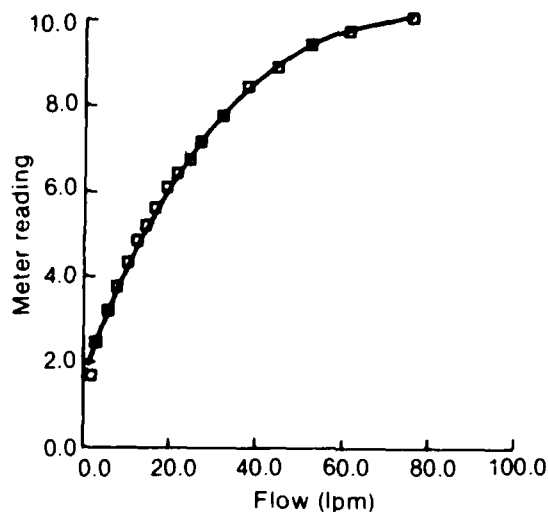


Figure 16. Dilution flowmeter calibration curve.

Since the temperature regulators had been on the market for many years and had performed well in other applications, it was decided to eliminate detailed thermal testing and use the manufacturer's specifications as an indication of temperature control. Even if the putative resolution of 0.002°C average deviation from the set point is exceeded by a factor of five, the oven still has a resolution of 0.01°C average deviation. This far exceeds the performance of previously available calibrators.

## 6. CONCLUSIONS AND RECOMMENDATIONS

The calibrator has been in the field for over two years as of the date of this report, and user response has been very favorable. The basic design is sound and has exceeded its performance goals. As far as future developments in this field are concerned, numerous improvements could be made:

(1) The fluidic oscillators can be made of smaller dimensions (with features as small as microns) to enable the permeation flowmeter to detect lower flowrates, thereby increasing the dilution ratio of the instrument.

(2) A microprocessor can be incorporated in the unit, thus eliminating any need for multiple temperature calibration curves and unit conversion. This would greatly simplify user operation.

(3) More elaborate electronic filtering of the microphone signals would extend the range of the flowmeters.

(4) Separate inputs for dilution and permeation flows would result in a more precise adjustment of the flow ratios. A less resistive input path would allow lower gas pressures at the inputs, which would simplify the sealing requirements on the system.

(5) A more efficient heat transfer path could be designed for initial temperature stability in the outer oven region. This would lead to increased response time for temperature adjustments.

(6) Bonded fluidic stacks would eliminate leaks between laminates.

(7) A new manifold could be designed which would be easier to fabricate and also allow easier access to the permeation chamber.

(8) A settling tank could be incorporated into the unit to help reduce the turbulence of the supply gases which lead to fluctuations in the flow measurement.

# NOMENCLATURE

$A_j$	= area of amplifier supply nozzle
$C_1$	= velocity profile constant
$b_s$	= width of supply nozzle
$f$	= frequency
$h_s$	= height of supply nozzle
$K$	= constant (see eq (9))
$Q$	= volumetric flow rate
$Q_j$	= volumetric flow rate of supply jet
$t_a$	= acoustic time delay (ac component)
$t_f$	= time constant of feedback channel (dc component)
$t_j$	= signal transport time of supply jet
$\bar{V}$	= average throughput velocity
$X_{sp}$	= nozzle-to-splitter distance
$\sigma$	= aspect ratio
$\tau$	= total period of oscillation

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APPENDIX A. STACKING ORDERS

APPENDIX A

ILLUSTRATIONS

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## APPENDIX A

The position and orientation of the fluidic laminates in a circuit are defined as the stacking order. For a more detailed description, see Joyce. Following are the two stacking orders for the fluidic oscillators employed in this device. Because of the absence of orientation information on the X-format laminates, a photocopy of the actual laminates used is given.

---

<sup>1</sup>J. W. Joyce, *Catalog of Fluidic C-Format Laminates*, Harry Diamond Laboratories, HDL-SR-83-2 (March 1983).


# APPENDIX A

Illustration A-1. Stacking order for permeation flowmeter.

Stacking order:	1	2	3	4	5	6
HDL drawing No. :	10042	10047	10047	10044	10033	10035
	7	8	9	10	11	12
	10034	10003 <sup>a</sup>	10034	10035	10033	10044

<sup>a</sup> $\sigma = 0.60$ ; 0.006-in. nozzle width amplifier

Illustration A-2. Stacking order for dilution flowmeter.

Notch position:							
							
Stacking sequence	Part number <sup>a</sup>	Notch	Quantity required	Stacking sequence	Part number <sup>a</sup>	Notch	Quantity required
1	5048A	A	1	21	5236A	H	1
2	5021A	B	1	22	630xx <sup>c</sup>	H	--
3	5021A	C	1	23	5236A	H	1
4	5217A	E	5	24	5237A	H	1
5	5414A	B	1	25	5239A	A	1
6	5239A	C	3	26	5018A	A	1
7	5021A	H	1	27	5239A	A	1
8	5011A	G	4	28	5237A	A	1
9	5048A	G	1	29	5236A	A	1
10	5239A	H	1	30	630xx <sup>d</sup>	A	--
11	5237A	A	1	31	5236A	A	1
12	5236A	A	1	32	5237A	A	1
13	630xx <sup>b</sup>	F	--	33	5239A	A	1
14	5236A	A	1	34	5018A	C	1
15	5237A	A	1	35	5053A	C	24
16	5239A	H	1	36	5118A	F	2
17	5118A	A	2	37	5042A	D	1
18	5018A	C	1	38	5043A	G	1
19	5239A	F	1	39	5022A	E	1
20	5237A	F	1	40	5022A	H	1
				41	Microphone/cover plate		

<sup>a</sup>Part numbers correspond to catalog numbers--see reference 1.

<sup>b</sup> $\sigma = 3.0$ , stage 1.

<sup>c</sup> $\sigma = 4.0$ , stage 2.

<sup>d</sup> $\sigma = 8.33$ , stage 3.

APPENDIX B. OPERATION MANUAL FOR HDL-CRDEC CHEMICAL CALIBRATOR

## APPENDIX B

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## B-1. INSTALLATION

The chemical calibrator requires a regulated, external supply of compressed air for flow input, as well as 120 Vac for the electrical system. A typical installation hookup can be seen in figure B-1. A well-regulated gas source is essential for optimal system operation.

In most cases, a compressed air tank with a regulator will suffice, but when operating at high flow rates ( $>30$  l/min) an external settling tank is recommended to supply a steady quiet flow source to the instrument. This tank will act as a filter to reduce disturbances present in turbulent flow paths.

For an unloaded system (exhausted into atmosphere) a supply pressure of 10 psi should be sufficient. However, if the output has a substantial load, it might be necessary to increase this input pressure in order to deliver enough flow for the system to operate over the entire range of the dilution flowmeter. Supply pressures greater than 50 psi are not recommended.

Under normal circumstances there is no need for any periodic maintenance to this instrument.

## B-2. OPERATOR INFORMATION

B-2.1 Temperature Controls

The oven chamber consists of two separate heating stages, an outer oven and an inner permeation oven, both with separate heat controls.

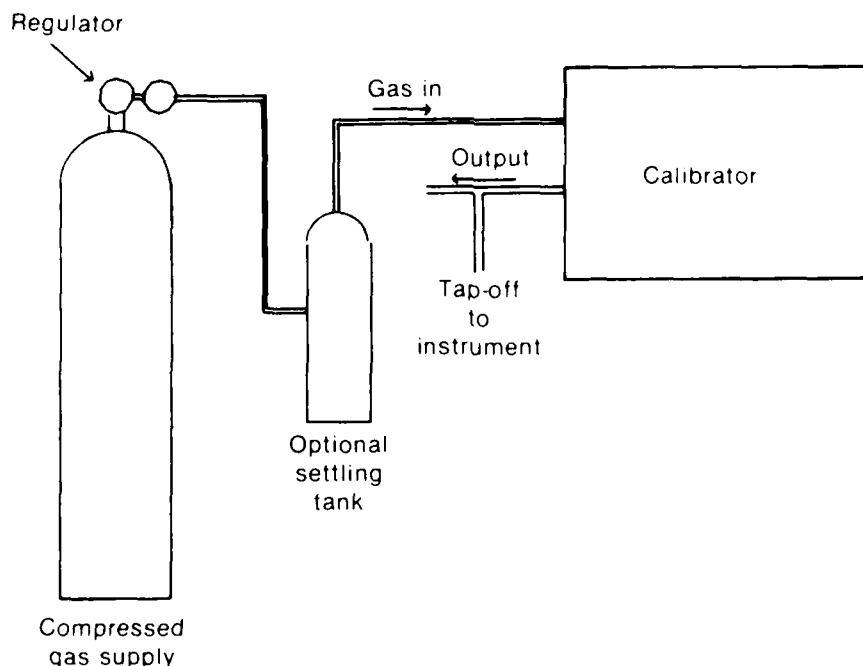


Figure B-1. Typical laboratory hookup.



## APPENDIX B

The front panel temperature display (see fig. B-2) indicates the temperature on the manifold block containing the permeation chamber. It is a tightly regulated control loop which is adjusted by the permeation oven controls, and is accurate to within  $\pm 0.01^\circ\text{C}$ .

There are two methods of adjusting temperature in the oven chamber. Adjusting the temperature setting of the outer oven causes more or less heat to be applied to the input gas heat exchanger (see fig. B-3). The basic idea behind this outer oven is to raise the temperature of the incoming gas to a point closer to the setting of the permeation oven, in order to achieve more accurate temperature regulation in the permeation chamber. It is a "helper" oven of sorts. Since the temperature regulation of the outer oven is relatively poor, it is recommended that the controls be set at least  $10^\circ\text{C}$  lower than the setting of the permeation oven, so as not to interfere with the operation of the permeation oven.

The second method of temperature adjustment is the permeation oven control. This instrument was calibrated at a permeation oven setting of  $35.0^\circ\text{C}$ , which corresponds to a permeation oven temperature of  $35.1^\circ\text{C}$  (display readout). This discrepancy in the setting and the actual temperature is due to the thermal time constant of the stainless-steel block used in the fabrication of the permeation chamber.

**IMPORTANT:** The warm-up time required to reach temperature equilibrium at  $35.1^\circ\text{C}$  is approximately 2-1/2 hours, at the minimum bandwidth setting (see sect. B-2.2).

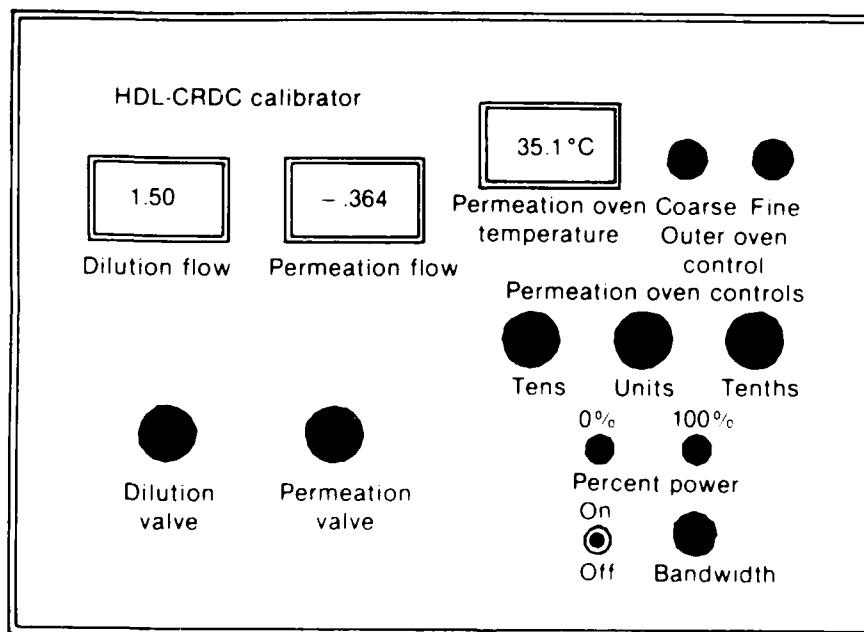


Figure B-2. Front panel controls.

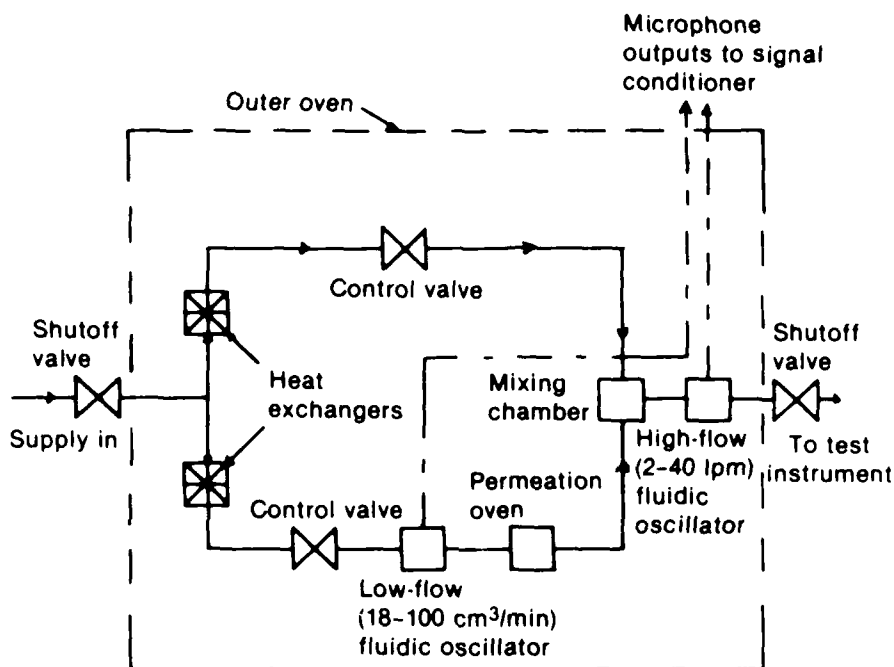


Figure B-3. Block diagram showing mechanical schematic of system.

The bandwidth control allows the accuracy of the temperature regulation in the permeation oven to be adjusted. The settings on the dial correspond to oscillation excursions around the temperature set point. Obviously, during use of this instrument, the lowest temperature excursion possible is desirable, so it is recommended that the bandwidth adjustment be kept at its minimum setting. A possible use for this bandwidth control might be to decrease total warmup time. By gradually decreasing the bandwidth from high to low during warmup, the total time required to attain regulated temperature will be decreased, but this requires supervision of the instrument, so it is usually easier to sacrifice savings in time for convenience in this case.

#### B-2.2 Flow Controls

There are two different methods for adjusting flow through the system: (1) increase the supply pressure to the system and (2) decrease the load on the system. Method 2 can be done either by adjusting the dilution or permeation valves (see fig. B-2) or by decreasing the load on the exhaust outlet of the instrument.

Flow is increased by turning the valves counterclockwise. Setting a desired permeation/dilution flow ratio is not elementary, because of the single input to the instrument. There is some isolation between the inputs to the two flowmetering devices, because of a small isolation chamber at the division of the input flow path (see fig. B-2). However, because of the large relative difference between the permeation flow ( $\text{cm}^2/\text{min}$ ) and the dilution flow (liters/min), complete isolation is not possible.

## APPENDIX B

Setting a desired flow ratio is at best a two-step process. First the dilution flow is set to the required value using the calibration information provided in section 3. When this setting is complete, then the permeation flow may be set.

IMPORTANT: Adjustment of the dilution flow, after the permeation flow has been set, will cause a change in the permeation flow. This is a result of the isolation problem discussed earlier. However, small adjustment of the permeation flow should have little to no effect on the dilution flow.

The dilution-flow display is the left LCD (see fig. B-2). When the valve is shut off, the display should read  $1.50 \pm 0.02$ . It is important to understand that flow less than the minimum flow range of the meter ( $<1.5$  l/min) may be passing through the valve before registering on the display.

The display is relatively slow in reacting to changes in the flow setting. Therefore, it is necessary to allow the display to come to a steady state before taking a reading. The worst-case response time is never more than 10 to 15 s from full off to full on.

The permeation-flow display is the right LCD (see fig. B-2). When the permeation valve is shut off, the display should read  $-0.364 (\pm 0.002)$ . The same situation exists between the no flow state (valve closed) and the minimum measurable flow state as on the dilution-flow side. There will be a noticeable click when the permeation valve is opened. This is a normal property of the valve and should not be of any concern. Slowly opening the valve causes the display to change to a positive value. The first good reading is  $+0.000$  as shown in the calibration information. The operator should practice with the valve to try to get some feel for the adjustability of the meter.

IMPORTANT: It is possible to overshoot the flow range of the permeation meter by allowing too much flow to pass through the valve. This will result in a wildly flickering permeation flow display reading. If this case occurs, the operator should gradually decrease the valve setting until the reading is steady. A good reading should have no more than  $\pm 0.001$  variation in the display.

If difficulty is encountered in setting the permeation-flow valve, it is probably because the input pressure to the instrument is too high, and the entire flow range may be passed over in a very slight turn of the permeation valve.

Two things can be done to alleviate this problem: (1) decrease the input pressure to the instrument or (2) increase the dilution flow. Both methods have the same effect, which is to reduce the pressure head at the input to the permeation valve, thereby making the valve easier to adjust.

### B-2.3 Permeation Chamber

The permeation chamber is integrated with the flowmeter manifold and is manufactured entirely from 316 stainless steel. It consists of a cylindrical chamber with a bottom inlet from the permeation flowmeter. This flow passes through the chamber and exits at the top, whereupon it travels through coiled stainless tubing and into the highly baffled mixing chamber.

The permeation chamber is sealed at the top by a bolted-down cover plate with an o-ring type of seal. The cover plate, as well as its mating surface at the top of the chamber, is manufactured from specially hardened tool-grade stainless steel to resist surface deformation caused by repeated opening of the chamber to reload. The o-ring is a 0.020 in. high by 15/16-in. OD gold o-ring which is seated in a groove at the top of the chamber.

IMPORTANT: This o-ring is not a reusable item. It must be replaced every time the chamber is opened. The chamber is made to accept a standard glass permeation tube with a porous bottom.

The cover plate is held down by four 1/2-in. nuts and washers mating to threaded studs on the chamber. The procedure for sealing is as follows:

1. Carefully place the permeation tube containing the agent into the chamber.
2. Place a new gold o-ring into the grooved seat at the top of the chamber.
3. Slide the cover plate over the studs very carefully so as not to dislodge the o-ring from its seat. It is essential that the o-ring remain in the seat for a seal to occur.
4. Place the washers on the studs and hand-tighten the four nuts to hold the cover plate in place.
5. Now the nuts must be torqued down in a diagonally opposed pattern to insure even force around the diameter of the o-ring. It is not necessary to torque down these nuts excessively. A good turn with a 1/2-in. nut driver is recommended for this operation.

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